PATENT SPECIFICATION

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NO DRAWINGS

1,118,876

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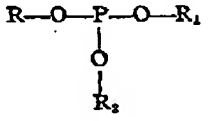
COMPLETE SPECIFICATION

Improvements in the Treatment of Synthetic Polyester Shaped Articles

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1., a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The treatment of shaped articles made from an essentially linear, crystalline polyester.

According to the present invention we provide a process for the treatment of shaped articles as hereinafter defined, made from a synthetic essentially linear, crystalline polyester containing an optical brightener, with a water-insoluble, co-crystallisable, polymeric compound containing at least one polyoxyalkylene group as hereinafter defined, the said crystallisable polymeric compound being applied to the surface of the shaped article and the treated, shaped article being thereafter subjected to thermal treatment at a temperature above 90°C., the said polymeric compound being applied in the form of an aqueous dispersion, the said aqueous dispersion containing an amount of a hindered phenol antioxidant as hereinafter defined equivalent to 0.2 to 1.0% by weight, based on the weight of the said polymeric compound, the said aqueous dispersion also containing a compound A in an amount equivalent to 0.2 to 2.0% by weight based on the weight of the polymeric compound, said compound A having the formula:



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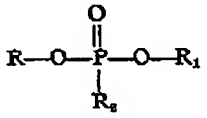

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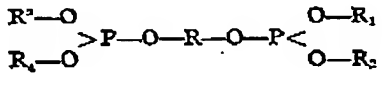

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where each of the groups R, R_1, R_2, R_3 and R_4 is an alkyl or aryl group or such that RH, R,H, R_2H, R_3H, and R_4H is a polyalkylene glycol, or being dipropylene glycol pentol triphosphite having the formula:—

(V)

An advantage of the process of our invention lies in the fact that by the use of it shaped articles of a polyester may have their surface properties modified to render the surface of the shaped articles hydrophilic and therefore wettable by water, and at the same time, the discolouration of the shaped articles, which occurs when they contain an optical brightening agent and are subjected to surface modification by a polymeric compound containing a polyoxyalkylene group at elevated temperature, is minimized.

The process of our invention is believed to be effective for use on shaped articles of polyesters containing any optical brightening agent suitable for producing an optically brightened polyester, whether by addition of the optical brightener to the starting materials or to the final polyester or by treatment during or after formation of the shaped article. The quantity of optical brightener present in the polyester may be any amount which is normally used in producing an optically brightened polyester and will vary with the effectiveness of the optical brightener and the degree of brightening desired.

The antioxidants herein referred to and which may be used in the process of our invention are those generally known as "hindered phenols", in which the hydroxyl group of the phenol is sterically hindered by one or more adjacent tertiary alkyl groups e.g. 2,4-dimethyl-6-α-methylcyclohexylphenol and 2,6-di-tertiary-butyl-4-methylphenol. Antioxidants containing two or more phenol groups connected by an alkylene bridge, e.g. bis(2-hydroxy-3-α-methylcyclohexyl-5-methylphenyl)-methane, or by a sulphur bridge as in bis(3-methyl-6-tert.butylphenol)-4,4'-disulphide are particularly effective.

Optical brighteners, which have been present in poly(ethylene terephthalate) fibres successfully subjected to the process of our invention are 1,2-bis(6-methylbenzoxazol-2-yl)ethylene, 2:5-bis(tert.butyl-benzoxazol-2-yl)thiophene, 2-cyano-4-naphthotriazolyl-4'-chlorostilbene and 3'-methylpyrazol-1'-yl-3-phenylcoumarin.

The essentially crystalline polyester may contain additives additional to the optical brightener, such, for example, as titanium dioxide.

By shaped article we mean a filament, fibre, fabric or film.

The shaped article to be treated may be in admixture with other materials, for example, in the form of a blend of polyester fibres with cotton fibres, wool or regenerated cellulose fibres.

Essentially linear crystalline polyesters which may be treated according to the process of our invention include fibre- and film-forming polyesters and copolyesters derived from poly(ethylene terephthalate), poly(tetramethylene terephthalate), poly-(1:4-bismethylenecyclohexane terephthalate), poly(ethylene naphthalene-2:6-dicarboxylate) and poly(ethylenediphenoxyethane-4:4'-dicarboxylate). Copolyesters may additionally contain, for example, adipate, isophthalate, sulphoisophthalate and paraoxybenzoate. The copolyesters may be derived from more than one glycol.

By co-crystallisable polymeric compound containing at least one polyoxyalkylene group, we mean a copolyester containing at least one polyoxyalkylene group and sufficient repeat units identical with those forming the crystalline portions of the polyester from which the article is made to confer crystallisability on the copolymer. Such a copolymer, when examined in the crystalline form, produces an X-ray diffraction pattern which contains reflections which are identical with the major reflections produced by the polyester in its crystalline form.

Good results have been obtained in the process of our invention when the polyoxyalkylene group is derived from a polyoxyalkylene glycol having an average molecular weight of 300 or above, and the Viscosity Ratio of the co-crystallisable polymeric compound containing at least one a polyoxyalkylene group, as measured in a 1% by weight solution in orthochlorophenol at 25°C. is not less than 1.1.

In order that our invention may be more fully understood, we give hereinafter some examples of methods in which our invention may be put into practice. These examples, in which all parts and percentages are, by weight, are not intended to limit the scope of our invention in any way.

Examples

Preparation of "Crystallisable Copolymeric Compound"

Dimethyl terephthalate (97 parts), ethylene glycol (78 parts) and poly(oxyethylene) glycol of average molecular weight 1540 (220 parts) were reacted together under ester-interchange conditions in the presence of 0.076 parts of calcium acetate $2H_2O$ as ester interchange catalyst and 0.220 parts by weight of 2:6-ditertiary butyl-4-methylphenol as antioxidant to protect the poly(oxyethylene) groups. When the theoretical amount of methanol for complete ester-interchange had been evolved, 0.1408 parts of a 24.8 percent solution of phosphorous acid in ethylene glycol was added followed by 0.0194 parts of antimony trioxide. The mixture was heated at 283°C. under polycondensation conditions until a high molecular weight polymer was produced. The product had Relative Viscosity 1.3 as measured in 1% orthochlorophenol at 25°C. and was crystalline.

A 4% by weight aqueous dispersion of the copolymer was prepared by gravel milling, and to the dispersion were added a hindered phenol which was bis(3-methyl-6-tertiarybutylphenol)-4:4'-sulphide in amount equal to 0.5% by weight of the weight of the copolymer and a phosphorous compound, also in amount equal to 0.5% by weight of the weight of the copolymer, which had one of the formulae given in the Table 1. The phosphorous compound and the hindered phenol were each added in the form of a 10% by weight solution in polyethylene glycol of average molecular weight 200.

Application of "Crystallisable Copolymeric Compound"

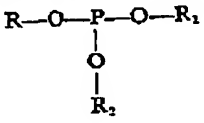
The dispersion of copolymer prepared as hereinbefore described, was padded onto poly(ethylene terephthalate) fabric which had been optically brightened with 1:2-bis(6-methyl-benzoxazol-2-yl)ethylene using a dyeing process, in such a manner that 3% by weight of the dispersed copolymer was retained on the fabric. The fabric was then heated in an oven at 200°C. for 5 minutes. After washing at 60°C. for a quarter of an hour with a washing powder, the reflectance spectra of the fabric was measured using a recording spectrophotometer fitted with a xenon lamp and "Pyrex" (Registered Trade Mark) glass filter to simulate sunlight; magnesium oxide was used as comparison standard. The whiteness (W) of the fabric was calculated using the equation $W = B - 0.75G + 75$, where B = total reflectance at 440 mμ and G = reflectance at 550 mμ.

TABLE 1

Phosphorous compound added	Whiteness of fabric	Weight loss on baking percent	Resistance of fabric megohms
Untreated fabric	109.0	—	8×10^6
Di-isodecylphenyl phosphite	107.5	0.0	5.2×10^3
Di-octadecylpentaerythrityl diphosphite	107.1	0.0	7.2×10^3
Tris-dipropyleneglycol phosphate	106.6	0.7	$10. \times 10^4$
Dipropyleneglycolpentol triphosphite	106.1	0.0	7.2×10^3
Tris-dipropylene glycol phosphite	105.5	1.8	6.3×10^5
None	104.5	2.5	1.42×10^4

WHAT WE CLAIM IS:—

1. A process for the treatment of shaped articles as hereinbefore defined, made from a synthetic essentially linear, crystalline polyester containing an optical brightener, with water-insoluble, co-crystallisable, polymeric compound containing at least one polyoxyalkylene group as hereinbefore defined, the said crystallisable polymeric compound being applied to the surface of the shaped article and the treated, shaped article being thereafter subjected to thermal treatment at a temperature above 90°C., the said polymeric compound being applied in the form of an aqueous dispersion, the said aqueous dispersion containing an amount of a hindered phenol antioxidant as hereinbefore defined equivalent to 0.2 to 1.0% by weight, based on the weight of the said polymeric compound, the said aqueous dispersion also containing a compound A in an amount equivalent to 0.2 to 2.0% by weight based on the weight of the polymeric compound, said compound A having the formula:



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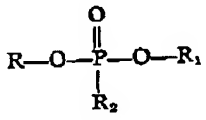

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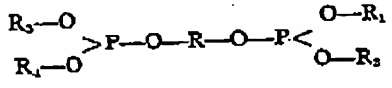

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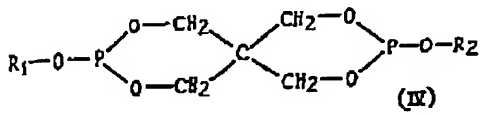

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where each of the groups R_1, R_2, R_3 and R_4 is an alkyl or aryl group or such that R_1H, R_2H, R_3H, and R_4H is a polyalkylene glycol, or being dipropylene glycol pental triphosphite having the formula:—

2. A process according to Claim 1 wherein the hindered phenol antioxidant contains two or more phenol groups connected by an alkylene bridge.

3. A process according to Claim 2 wherein the hindered phenol is bis(2-hydroxy-3-α-methylcyclohexyl-5-methylphenyl)methane.

4. A process according to Claim 1 wherein the hindered phenol antioxidant contains two or more phenol groups connected by a sulphur bridge.

5. A process according to Claim 4 wherein the hindered phenol is bis(3-methyl-6-tert.butylphenol)-4,4'-disulphide.

6. A process according to any one of the preceding claims, substantially as hereinbefore described with particular reference to the examples.

B. D. P. WETTERS,
Agent for the Applicants.

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